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## **DETAILED DESCRIPTION**

[Detailed Description of the Invention]

[The technical field to which invention belongs] this invention relates to the powdered manufacture method that the sharp powder of particle size distribution can be obtained, according to precision spraying of the liquid using the so-called micropump method. [0002]

[Description of the Prior Art] From the former, powder, such as ceramics and a metal, is used for various uses, such as a structural material, and, for the reason, the various powder manufacture methods are enforced. As the powdered manufacture method, methods, such as the method of grinding a raw material by wet or dry type and the so-called spray dry method of spraying a liquid, drying the drop by hot blast in an instant, and obtaining powder, are learned.

[0003]

[Problem(s) to be Solved by the Invention] although size [ these methods ] the place which is suitable for mass production method and is benefited industrially -- particle size distribution -- broadcloth -- (- it was difficult to obtain a uniform powder particle in) widely Therefore, it usually sifted out after powder manufacture, and although it was carrying out arranging particle size, there was a limitation in raising homogeneity more.

[0004]

[Means for Solving the Problem] The place which this invention is made in view of the above technical problem, and is made into the purpose is to offer the powdered manufacture method that the sharp powder of particle size distribution can be obtained. That is, after according to this invention breathing out the liquid used as the precursor of ceramic material and forming a minute drop, manufacture method \*\* of the powder characterized by obtaining the ceramic powder to which 90% or more of particle exists [ a secondary particle diameter / the particle size distribution ] to less than \*\*10% to a mean particle diameter by 10-180 micrometers is offered by heat-treating this minute drop.

[0005] In this invention, heat-treating is desirable, after carrying out the two or more kind regurgitation of the liquid used as the precursor of ceramic material, carrying out collision coalesce of the drops in flight / suspension process and making a minute resultant generate. Moreover, it is desirable to perform the regurgitation of a liquid by the micropump method in this invention, and, as for this micropump method, it is desirable that it is the piezo-electric type liquid regurgitation method which is made to drive a piezo electric crystal and carries out the regurgitation of the liquid. As this piezo-electric type liquid regurgitation method, furthermore, specifically The pump section in which two or more liquid pressurized rooms corresponding to a hole were prepared is joined, two or more nozzles which make a liquid inject -- the nozzle section in which the hole was prepared -- receiving -this nozzle -- By making a part of wall of this liquid pressurized room transform by piezo-electricity / electrostriction element, and making this liquid pressurized room produce a pressure the liquid supplied to this liquid pressurized room -- the aforementioned nozzle -- it is the fluid injector it was made to make inject from a hole, and it is desirable from excelling in chemical resistance and thermal resistance to use the fluid injector which comes to constitute the aforementioned nozzle section and the pump section from zirconia ceramics [0006]

[Embodiments of the Invention] After the manufacture method of the powder of this invention breathes out the liquid used as the precursor of ceramic material by the so-called micropump method and forms a minute drop, it is heat-treating this minute drop, and obtains ceramic powder with sharp particle size distribution.

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[0007] As a micropump method used by this invention, the method learned for ink-jet record etc. from the former is employable. Specifically, an electric charge control system, an electric machine conversion method, an electric thermal-conversion method, an electrostatic suction method, etc. can be held. An electric charge centrol system synchronizes excitation and oscillation in frequency, and makes a minute drop former the drop which is carrying out continuation in on with a pressure. This drop is electrified, it controls by the deflecting electrode, and the regurgitation and the non-regurgitation are controlled. An electric machine conversion method is a system which an element with similar functions, such as a piezoelectric device, is vibrated, and a drop is atomized using the energy which it has, and is made to breathe out.

[0008] An electric thermal-conversion method generates a foam in liquid by the heater element, and makes a drop breathe out by the pressure. Moreover, an electrostatic suction method makes a liquid attract and breathe out by the electrostatic force.

[0009] In this invention, it is desirable to use the piezo-electric type liquid regurgitation method which is an electric machine conversion method with which a discharge pressure is obtained, without heating a liquid among the above-mentioned micropump methods. As opposed to the nozzle section in which the hole was prepared and two or more nozzles which make a liquid inject as concrete equipment -- this nozzle -- by joining the pump section in which one or more liquid pressurized rooms corresponding to a hole were prepared, making a part of wall of this liquid pressurized room transform by piezo-electricity / electrostriction element, and making this liquid pressurized room produce a pressure the liquid supplied to this liquid pressurized room -- the aforementioned nozzle -- it is the fluid injector (drop regurgitation equipment) it was made to make inject from a hole, and the equipment which comes to constitute these nozzle sections and the pump section from zirconia ceramics is desirable

[0010] An example of this fluid injector is shown in <u>drawing 1</u>. The light-gage plate-like nozzle plate 13 in which the hole 12 was formed is formed by the green sheet of zirconia ceramics. <u>drawing 1</u> -- setting -- the nozzle of plurality [ section / nozzle / 11 ] -- on the other hand, the pump section 21 Similarly the spacer plate 25 in which two or more window parts 28 were formed, and the lock out plate 23 which puts on one spacer-plate 25 side, and covers a window part 28 are formed by the green sheet of zirconia ceramics, respectively, and the laminating of the whole is carried out, and it really calcinates and is constituted. In addition, the liquid input 16 is formed in the lock out plate 23. And on the superficies of the lock out plate 23, the piezo-electricity / electrostriction element 22 which consists of the lower electrode 31, piezo-electricity / electrostriction layer 32, and an up electrode 33 are formed.

[0011] the nozzle which the liquid with which it filled up in the cavity 15 when piezo-electricity / electrostriction layer 32 would deform and the capacity of the cavity (liquid pressurized room) 15 in which the window part 28 was covered and was formed would decrease, if electric field arose between the up electrode 33 and the lower electrode 31 according to the above fluid injectors opens for free passage to a cavity 15 -- it is injected from a hole 12

[0012] <u>Drawing 2</u> shows other examples of a fluid injector, and shows the composition with which the so-called pump section and the so-called nozzle section were shared. That is, the pump section 21 forms the spacer plate 25 in which two or more window parts 28 were formed, the lock out plate 23 which puts on one spacer-plate 25 side, and covers a window part 28, and the substrate plate 27 which puts on the another side side of a spacer plate 25, and covers a window part 28 by the green sheet of zirconia ceramics, respectively, is really calcinated and is constituted, and -- this example -- a part of spacer plate 25 -- a nozzle -- it is made for pump section 21 the very thing to also have a nozzle function by forming a hole 12 and the liquid input 16

[0013] As mentioned above, in this invention, even if a fluid injector is the case where the liquid of ceramic material precursors, such as an acetone system and a hydrochloric-acid system, is used since the whole of the component consists of zirconia ceramics for example, it can be applied and is excellent also in chemical resistance, thermal resistance, and toughness.

[0014] In this invention, the liquid used as the precursor of ceramic material is breathed out using the fluid injector of the above micropump methods, a minute drop is formed, this minute drop is heattreated, and desired ceramic powder is obtained. Thus, since the liquid is breathed out using the fluid injector of a micropump method, if this minute drop is heated, it is minute powder and the particle size distribution can obtain the ceramic powder of a very sharp uniform particle size. Specifically, particle size is 10-180 micrometers, and particle size distribution can obtain the ceramic powder with which 90% or more of particle exists to less than \*\*10% to a mean particle diameter.

[0015] The usual heating means can be used as the heat-treatment method of a minute drop, for example, dryness, the method of carrying out pulverization, etc. can use a proper means for a minute drop in an instant according to a use by hot blast like the method of spraying a minute drop into a

heating furnace, and drying and heat-treating it, and the so-called spray dry method. Moreover, in this invention, although it is a liquid used as the precursor of ceramic material as a target liquid, the slurry which carries out predetermined concentration content for example, of the ceramic material is included in this.

[0016] As mentioned about though it explained heating this with the mide comp method about this invention after injecting a liquid, it is also possible to prepare two kinds of equipment which injects a liquid with a micropump method, to make the minute drop injected from each equipment collide mutually, and to form a desired particle. For example, by injecting two kinds of liquids which react mutually, making them collide, and making both drops react, the resultant of short reaction time is also stabilized and can be obtained. Moreover, since the amount of liquids made to collide is easily controllable, a reacting weight is arbitrarily controllable. Furthermore, since a mutual collision speed of two kinds of liquids and an amount are controllable, it also becomes possible particle size, a particle shape, and to set up arbitrarily.

[Example] Next, a concrete example explains this invention further.

(Example 1) The powder manufacturing installation shown in <u>drawing 3</u> was used, the drop regurgitation equipment 40 of the structure which shows the ethyl alcohol solution of a zirconium chloride in drawing 1 -- using -- electromagnetism -- a shutter 41 -- heating -- business -- the drop which sprayed intermittently and was sprayed in the heating furnace 43 into the heating furnace 43 made from a quartz equipped with the external heater 42 was dried and pyrolyzed, and the zirconia ceramic powder A was obtained As for an air inlet and 45, 44 are [ an exhaust port and 46 ] particle recovery boxes among drawing. The obtained zirconia ceramic powder A was 20 micrometers of mean particle diameters, and the particle size distribution was a uniform thing by which 92% of particle goes into \*\*10% of range to this mean particle diameter.

[0018] (Example 2) The powder manufacturing installation shown in drawing 4 was used the ethyl alcohol solution of a zirconium chloride -- from drop regurgitation equipment 40a -- moreover, the ethyl alcohol solution of a sodium hydroxide -- the electromagnetism from drop regurgitation equipment 40b -- it arranges and controlled and breathed out as a minute drop so that collision coalesce might be carried out during each suspension through Shutters 41a and 41b, and collision coalesce was carried out in the air Then, it dried and pyrolyzed in the heating furnace 43, and the end of zirconia mixed powder was obtained. By the reaction inside the drop which coalesced, since the microcrystal of a zirconium hydroxide generated and the zirconia generated by the dehydration by heating after that, as compared with the example 1, the particle more detailed [a primary particle] and uniform was able to be obtained.

[0019] (Example 3) The powder manufacturing installation shown in drawing 5 was used. With this equipment, in order to spray two kinds of liquids, two sorts of drop regurgitation equipments 50 and 51 were used. In addition, 52 is a tank, 53 is the inlet of the liquid to a tank and 54 is output port. It sprayed into the ethyl alcohol solution of the sodium hydroxide which stored the ethyl alcohol solution of aluminum ethoxide in the tank 52 using the drop regurgitation equipment 51 of the structure shown in drawing 3 from the drop regurgitation equipment 50 of the structure which shows the ethyl alcohol solution of a zirconium chloride in drawing 2, and was made to react into the liquid concerned. The obtained resultant was transported to the liquid/separator which is not illustrated after that, is separating a resultant and making it decompose thermally there, and obtained the mixed-powder end of a zirconia particle and an alumina particle.

[0020] In this example, by carrying out the conte rule of the discharge quantity of the drop regurgitation equipments 50 and 51 suitably, it is a desired mixing ratio and the mixed-powder end of a zirconia particle and an alumina particle it was moreover mixed uniformly was able to be obtained. This powder is very suitable from the point which it faces producing an alumina / zirconia compound ceramics, and is made to form into distributed composite uniformly. In addition, like examples 1 and 2, like an example 3, maintenance of a drop configuration becomes easy and the method of solidifying by heating in the air has the advantage of being hard to produce coalesce at a separation dryness process, as compared with the method of carrying out postheat-treatment solidification which solidified in the liquid.

[0021]

[Effect of the Invention] As explained above, according to the manufacture method of this invention, the powder of a sharp uniform grain size of particle size distribution can be obtained. Moreover, when the fluid injector of the piezo-electric type liquid regurgitation method which constituted the nozzle section and the pump section from zirconia ceramics is used, it excels also in chemical resistance, thermal resistance, and toughness.